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Purposeful scientific research as a pillar of sustainable development

Green Synthesis of Nickel Oxide Nanoparticles Using Peppermint Extract: An Advanced Structural and Spectroscopic Study

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ABSTRACT

In this study, an eco-friendly approach for synthesizing nickel oxide nanoparticles (NiO NPs) was developed in accordance with green chemistry principles. Iraqi peppermint (Mentha sp.) extract was employed as a natural reducing and stabilizing agent. The synthesis involved reacting nickel nitrate with the plant extract under controlled thermal conditions, resulting in the formation of nanoparticles without the use of hazardous chemical reagents. Extensive characterization of the synthesized sample was performed using UV Var, XRD, SEM and EDS techniques. UV spotter showed an absorption peak at 720 Nm, which confirmed the synthesis of the nanomaterial. According to JCPDS card number 44-1159, XRD analysis revealed a pure faceted cubic (FCC) crystalline structure without evidence of secondary phases and diffraction stops. SEM images indicated a porous morphology with irregular nanoparticle -aglomerations, while the EDS exam validated the presence of nickel and oxygen as primary elements with nuclear positions showing high processing and structural unit. The findings clearly emphasize the potential of green synthesis, where locally available plant extracts act as a reducing agent to achieve nanomaterials with favorable properties. This approach is not only effective but also cost -effective and environmentally sustainable.

Keywords: Green synthesis, Morphpological characterization, Nickel oxide nanoparticles, Peppermint extract, Spectroscopic characterization.

Introduction

Nanotechnology has made rapid progress in recent years due to the potential to offer practical solutions to environmental challenges, especially water -contaminated with heavy metals. Among the promising materials stand out nickel -nanoparticles (nine -nps) due to their distinctive physical -chemical properties, including elevated surface area, thermal and chemical stability and remarkable catalytic activity (1). These properties make them high quality candidates for adsorption processes and catalytic surface reactions in contaminated water systems [1–3]. Although conventional procedures for synthesizing nanomaterials have proven efficient in terms of yield, they often rely on poisonous reagents and harsh reaction conditions, raising serious environmental and health concerns [4]. In response, the principle of green chemistry has emerged as an alternative approach, aiming to formulate sustainable and environmentally friendly synthesis routes that utilize natural plant-based sources rich in bioactive compounds capable of diminishing metal ions and stabilizing nanoparticles [5–6]. within the various plants explored, peppermint (Mentha sp.), locally known as “Botnj”, stands out as a promising yet underutilized for the synthesis of NiO NPs.

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Peppermint extract contains a diverse range of supplier active compounds such as carvone, limonene, rosmarinic acid, flavonoids, and tannins, all of which have been shown to act as potent reducing and stabilizing agents in green synthesis processes [7–8]. modren studies have demonstrated that this plant-based approach not only minimizes the environmental impact of the preparation but also enhances the structural and interface characteristics of the resulting nanoparticles in terms of size, distribution, and colloidal stability [9,11]. furthermore, the phenolic and flavonoid compounds present in peppermint isolate are believed to play a dual role by precisely regulating the nanoparticle growth process and providing long-term steadiness when applied in complex aqueous systems, including those contaminated with heavy metals or petroleum-derived pollutants [12–13].

Materials and Methods

Chemicals used

Nickel nitrate hexahydrate ($Ni(NO_3)_2 \cdot 6H_2O$) was used as the origin of nickel ions and was obtained at high purity (≥99%) from a reputable provider specializing in laboratory-grade chemicals. Peppermint (Mentha sp.) extract was prepared from recenyly havested leaves collected locally from clean agricultural areas in Iraq. The leaves were thoroughly washed with distilled water to eliminate dust and impurities, then air-dried at room temperature away from direct solar rediation. Afterwards, they were ground employing an electric grinder to obtain a fine powder.

Preparation of Plant Extract

To prepare the aqueous extract, 10 g of powdered peppermint leaves were accurately weighed and boiled in 100 mL of distilled water for 20 minutes under a glass cover to minimize the loss of volatile components. After boiling, the mixture was allowed to cool to room temperature and subsequently filtered through Whatman No. 1 filter paper. The resulting clear filtrate was used immediately for the synthesis process.

Green Synthesis of NiO Nanoparticles

A solution of nickel nitrate (0.01 mol/L) was first prepared in distilled water and then combined with the peppermint extract in an equal (1:1) volume ratio. The resulting mixture was magnetically stirred at 70 °C for 90 minutes. During the reaction, the color gradually shifted from pale green to deep brown, signifying the generation of nickel oxide nanoparticles. This color transformation corresponds with observations reported in earlier studies, where such visible changes are regarded as reliable evidence of a successful green (chlorosynthetic) synthesis process [6].

Separation and Purification of Nanoparticles

The mixture was then centrifuged (6000 rpm for 15 min) to separate the nanoprecipitate. The precipitate was filtered off and washed three times successively with distilled water and ethanol to be freed from the impurities and remaining unreacted organics. The obtained precipitate was dried in an oven at 80°C for 12 h to obtain a homogeneous nickel oxide nanoparticle powder.

Sample Preparation for Grinding and Storage

Upon drying, the material was carefully ground with a ceramic mortar and pestle, aiming at a fine powder with a narrow particle size distribution and a homogenous structure. This powder was kept dry in a dark, sealed glass container waiting for further crystallographic and spectroscopical study to keep the powder stable and to obtain the highest purification degree.

Results and Discussion

Results

This chapter describes and discusses the results of the experiments on ecofriendly synthesis of nickel oxide nanoparticles (NiO NPs) by using Mentha sp. Using (peppermint) extract natural reducer and stabilizer.

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A combination of spectroscopic and structural techniques was applied to prove the successful formation of the nanoparticles and to assess their key physicochemical properties

UV-Vis Analysis of Nickel Oxide Nanoparticles

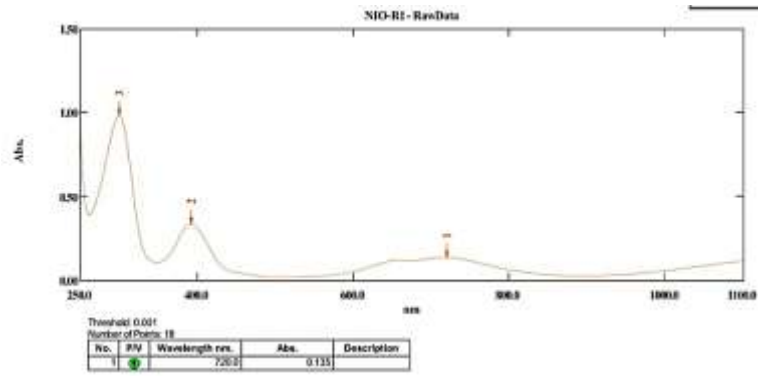


Fig. 1: The UV-Vis absorption spectrum of the nickel oxide nanoparticles synthesized using peppermint extract was recorded in the wavelength range of 250-1100 nm

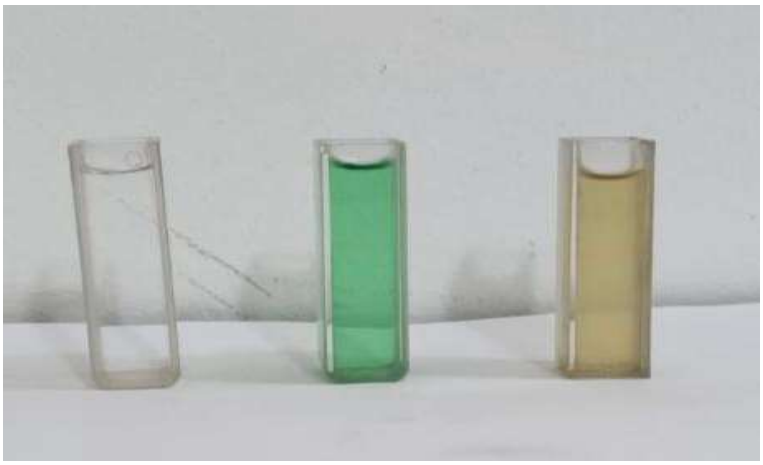


Fig. 2: Illustrative image of the samples used in the spectroscopic analysis, from left to right: the blank (non-ionic water), the NiO nanoparticle solution after the reaction, and the raw peppermint leaf extract before the reaction

XRD Analysis of Nickel Oxide Nanoparticles

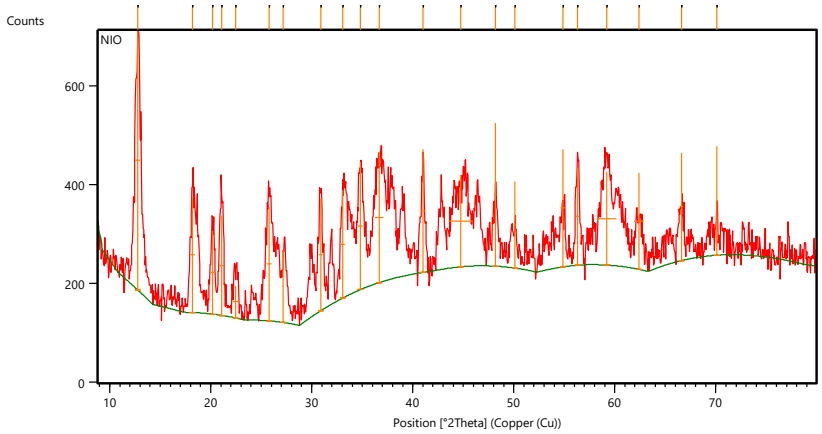


Fig. 3: X-ray diffraction (XRD) pattern of the NiO nanoparticle specimen prepared applying peppermint extract, recorded using Cu Kα radiation (λ = 1.5406 Å), showing the primary peaks corresponding to the (111), (200), and (220) crystal planes

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Table 1: Crystallographic parameters derived from XRD pattern of NiO nanoparticles synthesized using peppermint extract.

Peak No.	2θ (°)	Relative Intensity (%)	Miller Indices	JCPDS Reference
1	37.24	100.00	(111)	44-1159 (NiO)
2	43.30	49.58	(200)	44-1159 (NiO)
3	62.93	36.57	(220)	44-1159 (NiO)

Microstructural and Elemental composition Analysis of Nickel Oxide Nanoparticles Using SEM-EDS

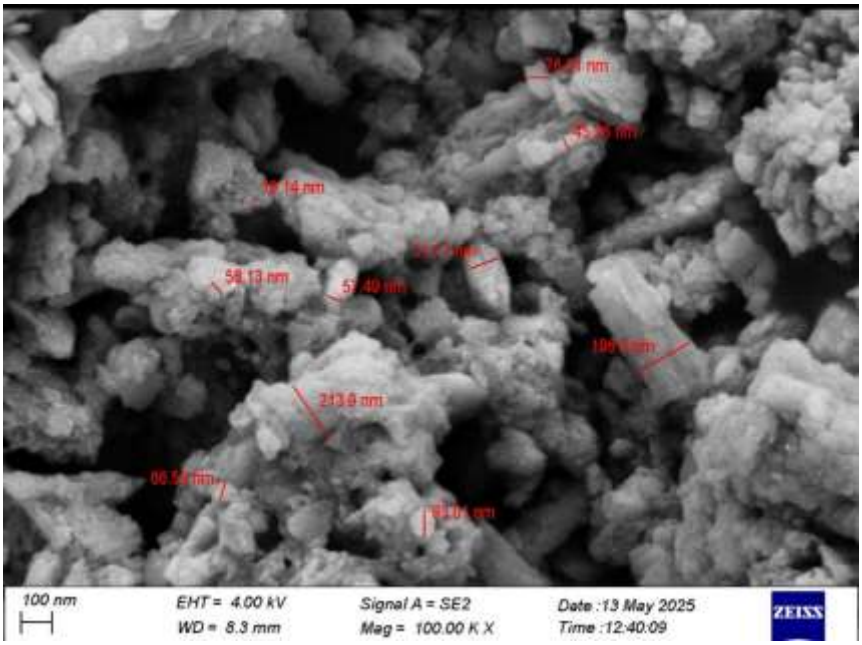


Fig. 4: The SEM micrograph of NiO nanoparticles synthesized using peppermint extract illustrates irregularly formed agglomerates with distinctly porous morphology, The particles demonstate a size distribution varying from approximately 45 to 213 nm, thereby validating the successful fabrication of nanostructures with heterogeneous dimensional characteristics

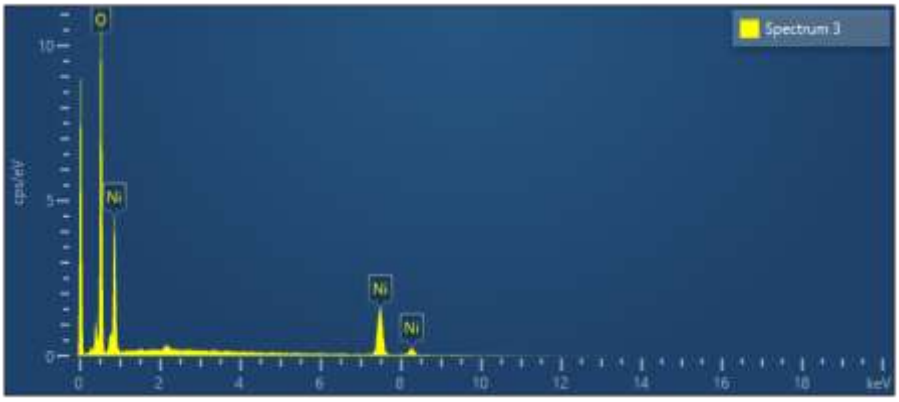


Fig. 5: The EDS spectrum of NiO nanoparticles synthesized using peppermint extract displays distinct characteristic peaks associated with nickel (Ni), thereby verifying the elemental composition and confirming the presence of nickel with a elevated degree of purity

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Discussion

UV-Vis Analysis of Nickel Oxide Nanoparticles

The synthesized nanoparticles were analyzed using UV–Visible (UV–Vis) spectroscopy within the wavelength range of 250–1100 nm. As shown in Figure 1, the sample exhibited a distinct absorption peak at 720 nm with an absorbance value of 0.135, confirming the successful formation of NiO nanoparticles. The presence of this peak corresponds to electronic transitions associated with the nanostructured nickel oxide, specifically the transition from Ni^{2+} ($3d^8$) to $3d^74s^1$, which is a characteristic feature of nanocrystalline semiconducting NiO materials [14-15].

Studies show that plant-based nickel oxide nanoparticles fall within the absorption range between 620 and 760 nm. The precise wavelength position varies depending on many factors including particle size, morphological characteristics and type of phytochemical components present in the plant extract during the synthesis [16,18]. The emergence of this absorption band gives initial confirmation for the successful green synthesis of NiO nanoparticles via peppermint extract because it indicates a similarity with the general spectral shifts arising from the nucleation and growth of such nanoparticles. Furthermore, with no other meaningful absorption peaks present, a high degree of phase purity can be inferred, indicating that the nanostructures should have low levels of organic residuals or secondary by-products. Such observations are consistent with those reported by Rashid et al. (2024) and Altaweel et al. First and foremost, smooth virulent well-defined absorption spectra of plant-derived NiO nanoparticles are indicative of uniform and homogeneous nanostructural development (Harshavardhan et al. 2023). [19–20].

XRD Analysis of Nickel Oxide Nanoparticles

In this study, nickel oxide nanoparticles (NiO NPs) were synthesized from peppermint extract, using a natural reducing agent in line with green chemistry principles. X-ray diffraction (XRD)) was used to characterize the crystalline structure and purity of the synthesized sample. As depicted in Figure 3, the sharp and well-defined peaks in the diffraction pattern at angles of $2\theta = 37.24°$, $43.30°$, and $62.93°$ were assigned to the (111), (200), and (220) planes, respectively. These peaks match closely with the standard JCPDS card No. 44-1159 for NiO, verifying the formation of a pure, face-centered cubic (FCC) phase without detectable impurities or secondary phases [21-22]. These findings demonstrate that the peppermint extract effectively reduced Ni^{2+} ions and stabilized the resulting nanoparticles. This efficiency can be attributed to the presence of phenolic and flavonoid compounds in the extract, which possess strong reducing and capping capabilities. Similar observations have been reported in recent studies, confirming the effectiveness of green synthesis using plant-derived extracts in producing NiO nanoparticles with well-defined crystalline structures and stable surface characteristics [23,25].

Microstructural and Elemental composition Analysis of Nickel Oxide Nanoparticles Using SEM-EDS

The surface morphology of the synthesized NiO nanoparticles was examined using scanning electron microscopy (SEM), as illustrated in Figure 4. Micrographs show a seemingly porous structure as a result of agglomeration of irregularly shaped nanoparticles - a unique feature evoked through words. Such a structure shows a high surface area, which is very suitable for use in adsorption, catalysis and electrochemical cells, as reported in previous studies [26]. Furthermore, preliminary analysis was performed on the same sample using energy -spreading X -ray spectroscopy (EDS), as shown in Figure 5. Only the occurrence of nickel (nine) and oxygen (O) elements were observed in the spectrum, there were no detectable signals for other elements, indicating the configuration of chemical molecules and compounds. The elemental composition analysis revealed that the weight percentages of oxygen and nickel

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were 50.94% and 49.06%, respectively. On the atomic scale, oxygen accounted for 79.21%, while nickel represented 20.79%. The minor deviation from the expected stoichiometric ratio can be explained by the presence of surface-associated oxygen species or hydroxyl groups derived from the phytochemicals in the reducing plant extract. These functional groups are likely adsorbed onto the nanoparticle surface during the synthesis process [27]. Comparable observations have been described in recent research; for instance, Zhang et al. (2020) found that plant-assisted synthesis of nickel oxide nanoparticles commonly produces porous structures enriched in oxygen, a feature linked to strong interactions between phytochemical residues and the nanoparticle surface. Likewise, Wang et al. As indicated by (2019), The morphology, crystallinity scale, and other structural properties of the nanoparticles produced greatly depend on the nature of the plant extract and the conditions selected for synthesis [28].

Conclusion

This study verified the efficiency of peppermint extract as a natural reducing agent in the environmentally friendly synthesis of nickel oxide nanoparticles (NiO), aligning with the principles of green chemistry. The UV–Visible spectrum revealed a clear absorption band at 720 nm, confirming the successful formation of optically active NiO nanostructures. In addition, X-ray diffraction (XRD) analysis indicated that the synthesized NiO exhibited a well-defined face-centered cubic (FCC) crystalline structure, consistent with JCPDS card No. 44-1159, and showed no evidence of secondary phases or crystalline impurities. Scanning electron microscopy (SEM) images displayed a porous and irregular surface morphology, reflecting enhanced surface area and structural complexity of the nanoparticles. Moreover, energy-dispersive X-ray spectroscopy (EDS) analysis confirmed that nickel and oxygen were the dominant elements in the sample, supporting a high compositional purity and the absence of detectable contaminants. These results highlight the success of the plant -mediated approach to produce NIO with appropriate structure and surface functions for various environmental and technological applications. The results underscore the prospect of using locally accessible plant extracts, such as peppermint, for the economically viable and sustainable production of high-quality nanomaterials, facilitating the way for their deployment in environmental remediation, notably in the adsorption of pollutants from manufacturing wastewater.

Author Contributions

The author cdid the whole research independently. Oly the idea of the study was discussed with the supervisor , Dr. Ataallah.

Funding Statement

Not applicable.

Conflicts of Interest

The authors declare no competing interests..

Ethical Approval and Consent

Not Applicable

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